EPA APPROVED

U-02-RC

ISOTOPIC URANIUM IN BIOLOGICAL AND ENVIRONMENTAL MATERIALS

Contact Person(s) : Isabel M. Fisenne

APPLICATION

This procedure has been used to analyze soft tissue, vegetation, water, and air filter samples (Hindman, 1983; Sill and Williams, 1981; Welford et al., 1960).

Uranium from acid leached, dry-ashed and wet-ashed materials is equilibrated with ^{232}U tracer, and is isolated by anion exchange chromatography. The separated U isotopes are microprecipitated for α spectrometry.

SPECIAL APPARATUS

1. Ion exchange columns (see Specification 7.5).

2. Polyethylene dispensing bottles (see Specification 7.11).

3. Special apparatus for the microprecipitation of U are listed under the generic procedure, G-03.

* *Environmental Protection Agency - Guidelines Establishing Test Procedures for the Analysis of Pollutants, Under the Clean Water Act; National Primary Water Regulations and National Secondary Drinking Water Regulations; Methods Update, tentatively slated for approval, 66FR3466-3497, January 16, 2001.*

SPECIAL REAGENTS

1. Uranium-232 tracer solution - about 0.3 Bq g^{-1} of solution in a dispensing bottle.

2. Bio Rad AG 1-X4 (100-200 mesh), anion exchange resin (see Specification 7.4).

SAMPLE PREPARATION

A. Vegetation and soft tissue.

1. Dry ash the sample according to the method described in Procedure Sr-02-RC (see **Note 1**).

2. Weigh out 10 g of ash and transfer to a 400-mL beaker.

3. Add a weighed amount of ^{232}U tracer solution (~ 0.03 Bq) from the dispensing bottle (see **Note 2**).

4. Add 200 mL of HNO_3 to the beaker and evaporate slowly to dryness.

5. Add 25 mL of HNO_3 to the beaker. Repeat the acid addition and evaporation until a white residue is obtained. (**Note:** If silicious material is present, transfer the sample to a 100 mL platinum dish or a 100 mL Teflon beaker with HNO_3. Add 10 mL of HF to the vessel and evaporate to dryness. Repeat additions of 25 mL HNO_3 - 10 mL HF as necessary to volatilize the silica. Remove the HF by adding three successive 10-mL volumes of HNO_3 to the vessel and evaporate to dryness.)

6. Add 25 mL of HCl and evaporate to dryness. Repeat the acid addition and evaporation twice more.

7. Heat to dissolve the residue in 50-100 mL of 7N HCl.

8. Continue with **Determination**.

B. Water.

1. Evaporate the H_2O sample to a small volume.

2. Add a weighed amount of ^{232}U tracer solution (~ 0.017 Bq) from a dispensing bottle and evaporate slowly to dryness (see **Note 2**).

3. Add 50 mL of HNO_3 and evaporate to dryness. Add 25 mL of HNO_3 and evaporate twice more.

4. Dissolve the residue in 25 mL of HCl and evaporate to dryness. Repeat the HCl addition and evaporation.

5. Heat to dissolve the residue in ≤ 50 mL of 7N HCl.

6. Continue with **Determination**.

C. Air filters.

Cellulose filters:

1. Add a weighed amount of ^{232}U tracer solution (~ 0.017 Bq) from a dispensing bottle to the filter in a platinum dish and dry ash in an electric muffle at 550°C (see **Note 2**).

2. Dissolve the residue in HNO_3 and transfer to a 250-mL beaker.

3. Add 25 mL of HNO_3 and evaporate to dryness. Repeat the acid addition and evaporation twice more.

4. Dissolve the residue in 25 mL of HCl and evaporate to dryness. Repeat the HCl addition and evaporation twice more.

5. Heat and dissolve the residue in ≤ 50 mL of 7N HCl.

6. Continue with **Determination**.

Glass fiber filters:

1. Place the filter and a magnetic stirring bar in a 400-mL Teflon beaker. Add a weighed amount of ^{232}U tracer solution (~ 0.033 Bq) from a dispensing bottle.

2. Add 100 mL of HNO_3, mechancially stir while heating for 1 h. Reduce the solution volume to ~ 25 mL. Remove the stirring bar and rinse with H_2O.

3. Add 10 mL of HF and evaporate to dryness.

4. Repeat the 25 mL HNO_3 - 10 mL HF additions and evaporations as necessary to volatilize the silica.

5. Add 25 mL of HNO_3 to the beaker and evaporate to dryness. Repeat twice more.

6. Heat and dissolve the residue in 25 mL of HCl and evaporate to dryness. Repeat the HCl addition and evaporation twice more.

7. Dissolve the residue in ≤50 mL of 7N HCl.

8. Continue with **Determination**.

DETERMINATION

1. Pass the 7N HCl sample solution obtained during sample preparation through a prepared anion exchange column (see **Note 3**). Discard the column effluent.

2. Wash the column with 400 mL of 7N HCl. Discard the washings.

3. Elute the uranium with 200 mL of 1N HCl, collecting the eluate in a 250-mL beaker. Discard the resin.

4. Evaporate the eluate to near dryness.

5. Destroy any residual organic material with dropwise additions of HNO_3.

6. Evaporate the solution to dryness. Dissolve the residue in a few drops of HCl.

7. Convert the solution to the chloride with three 5-mL additions of HCl.

8. Add 1-2 mL of 1N HCl, prepared with filtered water (see Procedure G-03, Microprecipitation Source Preparation for Alpha Spectrometry). Cool to room temperature.

9. Continue the analysis under Procedure G-03, Microprecipitation Source Preparation for Alpha Spectrometry.

Notes:

1. Freeze-dried or wet tissue may be wet ashed directly in HNO_3. Proceed with Step 3 of **Vegetation and Soft Tissue**.

2. It is necessary to analyze reagent blanks with each batch of samples to correct the U results.

3. 20 mL of Bio-Rad AG1-X4, prepared according to Specification 7.4 are conditioned with 500 mL of 7N HCl.

LOWER LIMIT OF DETECTION (LLD)

Uranium Isotopes

Counter Efficiency	(%)	40
Counter Background	(cps)	3.33×10^{-6} for ^{238}U
		3.33×10^{-6} for ^{234}U
Yield	(%)	85
Blank	(cps)	3.33×10^{-6} for ^{238}U
		3.33×10^{-5} for ^{234}U
LLD (400 min)	(mBq)	0.23 for ^{238}U
		0.53 for ^{234}U
LLD (1000 min)	(mBq)	0.21 for ^{238}U
		0.48 for ^{234}U
LLD (5000 min)	(mBq)	0.065 for ^{238}U
		0.15 for ^{234}U

REFERENCES

Hindman, F. D.
"Neodymium Fluoride Mounting for Alpha Spectrometric Determination of Uranium, Plutonium and Americium"
Anal. Chem., 55, 2460-2461 (1983)

Sill, C. W. and R. L. Williams
"Preparation of Actinides for Alpha Spectrometry without Electrodeposition"
Anal. Chem., 53, 412-415 (1981)

Welford, B. A. , R. S. Morse and J. S. Alercio
"Urinary Uranium Levels in Non-Exposed Individuals"
Am. Ind. Hyg. Asso. J., 21 (1960)